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Report No. 5

# RESEARCH ON ELECTROCHEMICAL ENERGY CONVERSION SYSTEMS

Interim Technical Report

By

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## SUMMARY

This report describes work on two tasks related to improved electrochemical energy conversion systems for vehicle propulsion.

The first is related to the lithium-air cell, specifically the reduction of oxygen at a platinum electrode in 1 M $LiClO_4$ solution in propylene carbonate. Voltage scans in the anodic direction at 200 mv/second gave an oxygen reduction peak at -0.42 volts (versus S.C.E.). An anodic peak at -1.3 volts and a shoulder on a cathodic branch at -2.12 volts had not been observed in the less pure solutions. There is evidence of impurities even in these highly purified solutions.

The second task reported on deals with the mathematical treatment of battery data. The testing of a battery to simulate an 8-hour excursion at various load profiles is described. The treatment is extended to the simulation of vehicle propulsion wherein the battery is associated with a 30 kw prime power source.

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# I. INTRODUCTION

This is the fifth semi-annual report of research on high-energy electrochemical energy conversion systems. The overall program to this point has been divided into seven tasks:

1. Determination of open circuit potentials of a series of couples in various electrolytes, and at appropriate temperatures;

2. Determination of the reversibility of these couples;

3. Electrochemical studies of high-energy couples leading to evaluation of these couples as materials for construction of high-energy electrically rechargeable storage systems;

4. Determination of kinetic parameters and evaluation of the rate limiting factors of selected reactions of electrochemical couples at appropriate electrodes;

5. Determination of kinetic parameters of selected reactions at catalytic electrodes.

6. Investigation of ion transport processes in membranes and/or electrolytes at elevated temperatures, and

7. Mathematical analysis of performance characteristics of electrochemical energy conversion devices.

The first four semi-annual progress reports described work on Tasks 1, 2, 3, 4, 6, and 7. During this reporting period work continued on Tasks 4 and 7.

## II. TASK FOUR - OXYGEN ELECTRODE IN PROPYLENE CARBONATE

### A. Introduction

This is a continuation of the investigation of the oxygen reaction at a smooth platinum electrode in the solution 1M $LiClO_4$- propylene carbonate. The theoretical basis for this work has been discussed (1).

### B. Experimental

1. Purification of materials. Propylene carbonate was distilled at 3 mm Hg pressure in the apparatus previously described (2). This pressure was selected to lower the boiling point sufficiently to avoid thermal degradation, yet maintain adequate boiling point difference between propylene glycol and propylene carbonate (3). A first cut amounting to 25 percent of the total was discarded. Boiling point data observed are given in Table I.

Lithium perchlorate (G. F. Smith Co., trihydrate) used in this experiment was first recrystallized from water, then heated in vacuo to 80°C until the monohydrate was formed. The temperature was then raised to 220°C for several hours. The weight loss of the resulting material was sufficient to account for three moles of water being lost.

"Ultra-high-purity" nitrogen and "aviator's breathing" oxygen (Air Products Company) were passed through a drying tube containing phosphorus pentoxide just before entering the cell.

Solvents used in the oxygen determination experiments were reagent grade or redistilled.

2. Apparatus. The apparatus used has been described in previous reports (1, 2).

3. Procedure. The electrode was potentiostated at the initial voltage and gas passed into the solution through a fritted glass gas sparger for a

TABLE I

EXPERIMENTAL BOILING POINTS OF PROPYLENE CARBONATE

| Pressure (mm. Hg) | Head Temperature (°C) |
|---|---|
| 2.0 | 76 |
| 3.0 | 84 |
| 3.9 | 86.5-87 |
| 6.5 | 93 |

period of thirteen minutes. The gas flow was then stopped for two minutes to allow the solution to come to rest and the scan was initiated. At least three scans were made in the same manner before reproducibility was achieved. The first and second scans of each series always gave higher currents.

C. Results and Discussion

Scans were made at 100 mv/second and 200 mv/second in continuance of the work reported in the last report (3). The scans made at 100 mv/second showed no distinct difference between the oxygen-saturated solution and the nitrogen-saturated solution. The scans made at 200 mv/sec. are shown in Figure 1. An oxygen reduction peak occurred at approximately -0.67 volt versus the S.C.E. When the background was deducted, the peak was shifted to about -0.42 volts and amounted to about 38 microamps/cm$^2$ of geometric surface.

It may be observed in comparing this scan with those previously reported (3) that the currents obtained decrease markedly as the purity of the solution improves. The major part of the currents obtained were still apparently the result of impurities in the solution although in this experiment no peaks at -1.8 cathodic and -1.4 anodic corresponding to water or propylene glycol respectively were present. A feature of this scan not observed in previous runs was the shoulder on the cathodic branch at about -2.12 volts. Also on this run an anodic peak at -1.3 volts was present. These had not been observed previously, but were possibly masked by larger peaks from other impurities on scans made in less pure solutions.

In work reported by Toni, et. al. (4), the oxygen wave in nitrosodimethylamine solution using phenyltrimethylammonium hexafluorophosphate as supporting electrolyte occurred at about -1.0 volt compared with about -0.7 volt in this investigation. He showed by means of the relationship of peak current to scan rate that the mechanism of the reaction in his solution was an ECE type, i.e., a chemical step between two electrochemical steps.

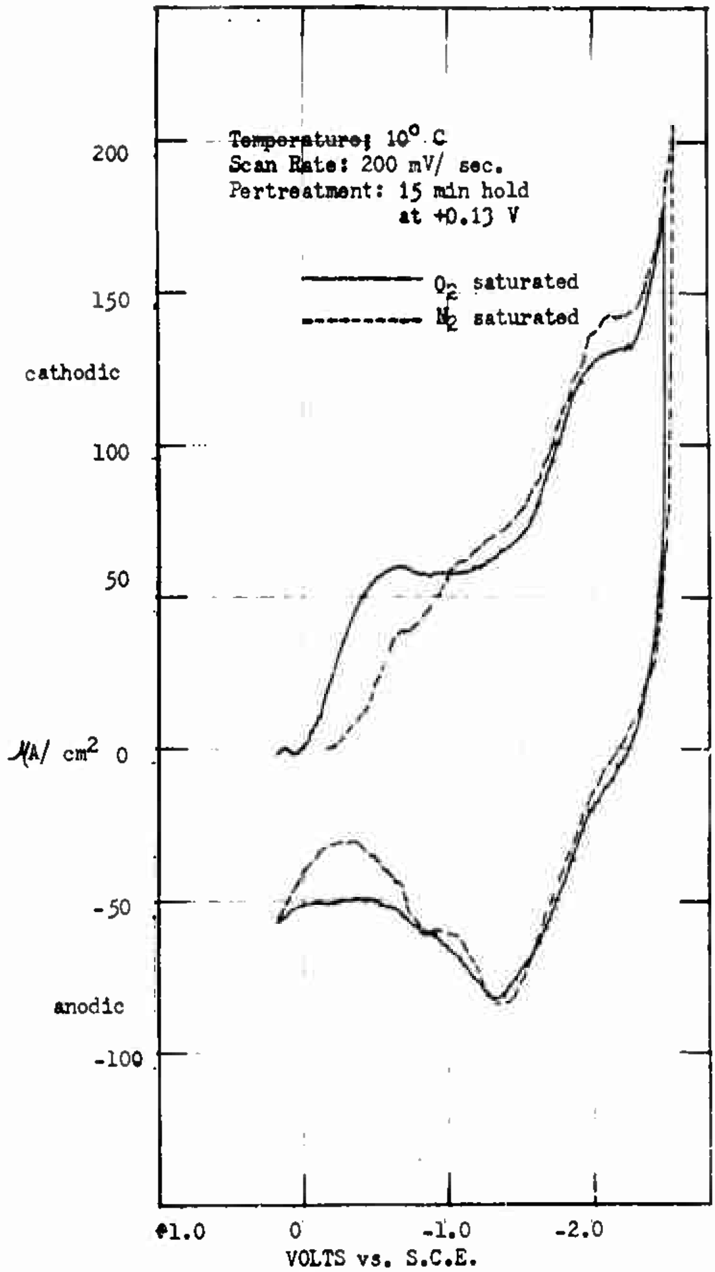


Figure 1. Voltage scans for Oxygen reduction in Propylene Carbonate, scan rate, 200 mV/sec.

He also suggested, but without supporting evidence, except that it would be expected to be consietent with his resulte, that there existed a reaction regenerating $O_2$ from the product of the firet charge-tranefer step. The amount of water preeent affected his resulte; increasing amounts of water resulted in higher peak currents for the first electrochemical step and less negative voltages for the second cathodic peak.

The major differences between Toni's solution and ours are simply these:

a) different solvent

b) different electrolyte

It would not be expected that differencee in viecosity or solubility alone would be responsible for our lower oxygen reduction currents, for Toni found a considerable portion of hie current wae due to adsorbed oxygen by his experimente using different potentiostatic holding times.

It muet be that there exieted eome subetance in our eolution which prevented oxygen adeorption onto the electrode. Thie could be one of three things: propylene carbonate, lithium perchlorate, or impurities. It cannot be determined without additional experimental work which of these three possibilities actually affected the results. This can be checked by (1) changing solvents; (2) changing supporting electrolyte, and (3) improving the purity of the solution still further.

The current levele obtained by Toni (4) were on the order of 1 milliamp/cm$^2$. Since these were peak voltametric currents it would be safe to assume steady-state currente in the range of two-thirds as much, or approximately 700 microamps/cm$^2$.

## III. TASK SEVEN - MATHEMATICAL ANALYSIS OF ELECTROCHEMICAL ENERGY CONVERSION DEVICES

### Battery Testing to a Random Load Profile

A. Objective

The ideal evaluation of a battery for a specific application is to test the battery under conditions identical to those under which it will be used. However the load profile of actual use conditions may be difficult to determine. For example, for a battery operated vehicle driving over an "average" terrain, a power vs. time curve would be nearly random in power requirements due to accelerations, stops, etc., and would be difficult to describe. This real time load profile could be in the form of Fig. 2 over an 8-hour driving period.

Over a period of time, say 8 hours, an estimate can be made of the total time spent at a given load and an average load profile determined. For the vehicle profile of Fig. 2 , the average load profile for 8 hours would be of a form similar to Fig. 3 where certain loads are more frequent than others. A load profile for battery testing can be created by selecting loads from the average profile and applying them to the battery for the indicated length of time. The results of such a test can be considered as an indication of how the battery would have responded in the real time profile, or if severe loads are selected, conclude that any battery performing satisfactorily in this test would also perform satisfactorily in real time profile. Whichever interpretation is used for the test results, the results will be affected by the length of time each load is applied and by the sequence of loads, e.g., the higher loads in the beginning or end of the cycle.

The test results of a battery will be biased by the particular sequence of loads selected, particularly if many cycles of the same sequence are

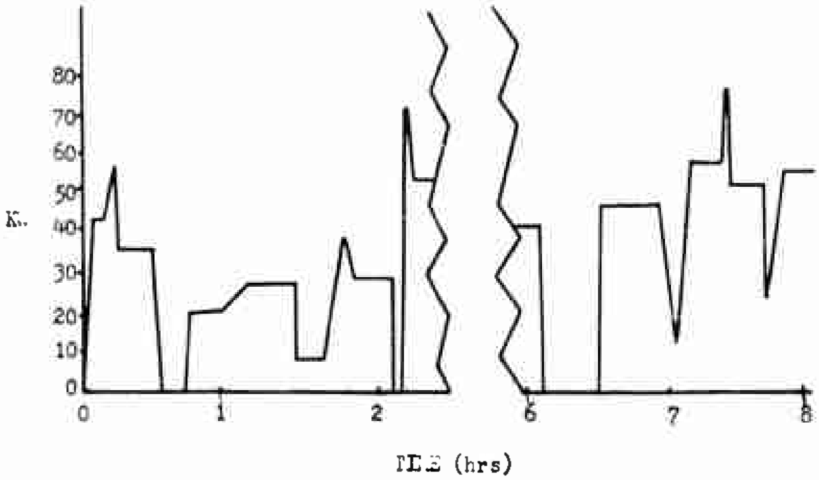


Figure 2. Load profile for a battery operated vehicle during 8-hour service.

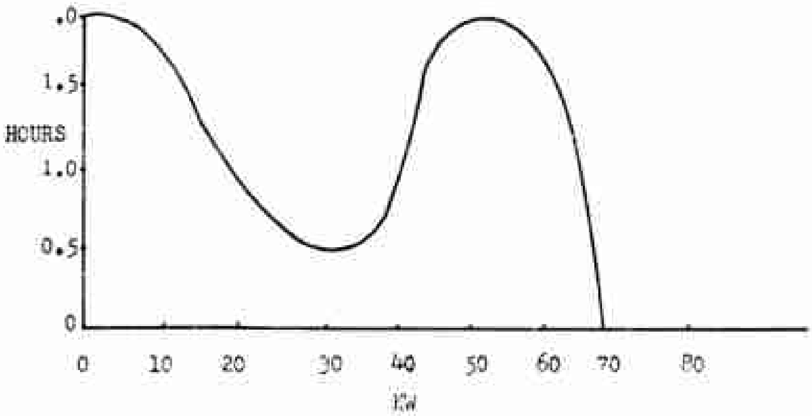


Figure 3. Average load profile for actual load profile given in figure 2.

applied. But this load sequence is abstracted from an average load profile and there are no criteria available for selecting one sequence over another. For instance, there is no justification for selecting a test profile composed of a high power step followed by a low power step followed by a medium power step. Likewise the length of time which a given load is applied is an estimate of an average and need not be a fixed number. Since there is no reason for selecting any given sequence, the most meaningful and useful data can be obtained by testing the battery under a variety of load sequences and durations of load application. It is possible to simulate this variety by Monte Carlo methods.

B. Discussion

A load profile and 8-hour discharge sequence postulated for a 50 KW vehicle which corresponds to the equivalent of four full power hours is: (5)

a) 50 KW for 1 hr

b) 25 KW for 1 hr

c) 75 KW for 1/3 hr

d) 45 KW for 2/3 hr

e) 10 KW for 2 hrs

f) Open Circuit for 2 hrs

g) 50 KW for 1 hr

Figure 4 is a graphical representation of this profile. However, repeated cycling of a battery to the same load profile may produce results unique to that particular test profile, and not a good indication of how the battery would behave in actual use. A variety of discharge sequences is needed to eliminate test artifacts peculiar to a given sequence.

The above load profile (Fig. 4) can be made more general by considering it to have been formed from the 8-hour average load profile of Fig. 5 which

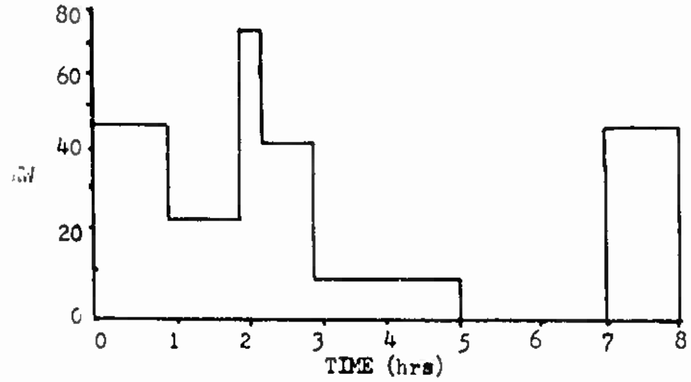


Figure 4. Actual load profile for a 50 KW vehicle on 8-hour duty

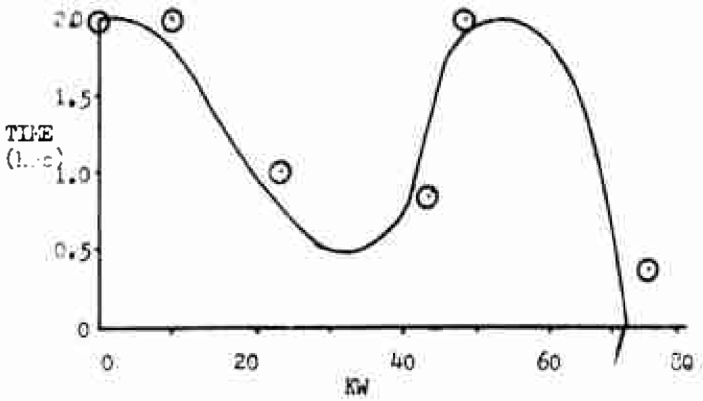


Figure 5. Average load profile for actual load profile given in figure 4

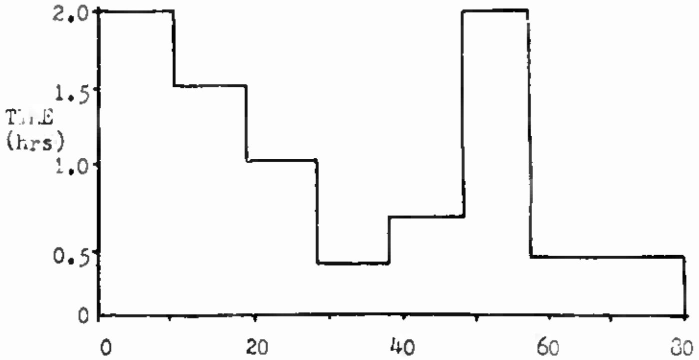


Figure 6. Stepwise approximation of load profile of figure 5

ie the data of reference 5 with the set sequence eliminated and expressed as a continuous curve.

Fig. 5 can be step-wise approximated into Fig. 6.

This is now in the form of an 8-hour average load profile similar to Fig. 3 and new discharge sequence can be selected. To generate a new sequence, the first load to be applied, the second load, etc. must be selected by some method. Since there is no justification for selecting any load preferentially, loads must be selected at random. Then, if the numbers [0, 1, 2, ---, 7] are assigned to correspond to the loads [0KW, 10 KW, 20 KW,--- 70 KW], a random selection of a number [0, 1, 2 ---, 7] will determine the first load of the discharge sequence. Similarly, a second random number is selected to determine the second load of the sequence, etc. If each load is to occur only once in an 8-hour discharge cycle, the number corresponding to that load must be excluded from succeeding selections once it has been chosen. A profile is completed when all eight loads have been applied. Thus, numerous discharge sequences can be generated which, if applied successively to a battery, eliminate any bias to the results due to any one discharge sequence.

Further generality can be added to the test profile if the length of time which a given load is applied is not held as a fixed number. The test profile is obtained from an average load profile which is an estimate of the length of time which a given load is applied over an 8-hour period. This length of time need not be the same for every 8-hour period but can be considered to be an average value of many 8-hour periods. Then, if many 8-hour periods are taken, the length of time a given load is applied will be a variable which has an average and is distributed about this average by some probability distribution. This will allow a given load to be applied for numerous time durations which will tend toward an average value.

For convenience, assume this probability distribution to be Normal (Gaussian). An average and a standard deviation ($\sigma$) are necessary to describe the time allotted for each of the eight loads mentioned above. The average time will be taken as that time for each load in Fig. 6. Since a standard deviation is not apparent from the average load profile, it may be arbitrarily assigned.

The selection of $\sigma$ must be such that reasonable time durations are obtained. For instance, if the average time for a given load is 2 hours it would not be expected that in any one 8-hour profile this time would be drastically different from 2 hours, but would usually be between 1.5 hours to 2.5 hours. If $\sigma$ is chosen to be one-eighth the average time for each load, reasonable time distributions are obtained.

Then, by the properties of the Normal distribution, in 96% of the 8-hour average load profiles, the length of time which a given load is applied will be within $[\text{Avg.} \pm 2\sigma]$. For the eight loads discussed above, the time values will be:

| Load (KW) | $[\text{Avg.} \pm 2\sigma]$ (hrs) |
|---|---|
| 0 | $2 \pm .5$ |
| 10 | $1.33 \pm .33$ |
| 20 | $1.0 \pm .25$ |
| 30 | $.33 \pm .08$ |
| 40 | $.67 \pm .17$ |
| 50 | $2 \pm .5$ |
| 60 | $.33 \pm .08$ |
| 70 | $.33 \pm .08$ |

Fig. 7 is a probability graph of the time distribution for these loads. It is read as: for any load, what is the probability that the load will be applied for a length of time less than or equal to some selected time. Then a length of time to apply a load will be determined by randomly selecting a two digit number [.00, .01, .02, ---, 1.0] to determine a probability value and read from Fig. 7 the length of time corresponding to that particular load. For instance, if the number selected to correspond to the load is 5 (50 KW) and the probability value selected is .25, a 50 KW load is applied for 1.84 hr as the first step of a discharge sequence. A new load and probability value is selected for the second step of the sequence, etc.

Thirty-five sets of random variables were chosen to determine five different test load profiles and are shown in Fig. 8. There is no noticeable pattern of occurrence of any particular load among the cycles.

The length of any cycle should be approximately eight hours. The time limits of the generated cycles are 7.12 hrs. and 8.36 hrs., the average of the five is 7.87 hrs. The average length of time each load is applied is:

| Load (KW) | Avg. Time (Hrs) |
|---|---|
| 0 | 1.97 |
| 10 | 1.40 |
| 20 | 1.01 |
| 30 | 0.32 |
| 40 | 0.62 |
| 50 | 2.09 |
| 60 | 0.31 |
| 70 | 0.32 |

which are approximately the values of Fig. 6.

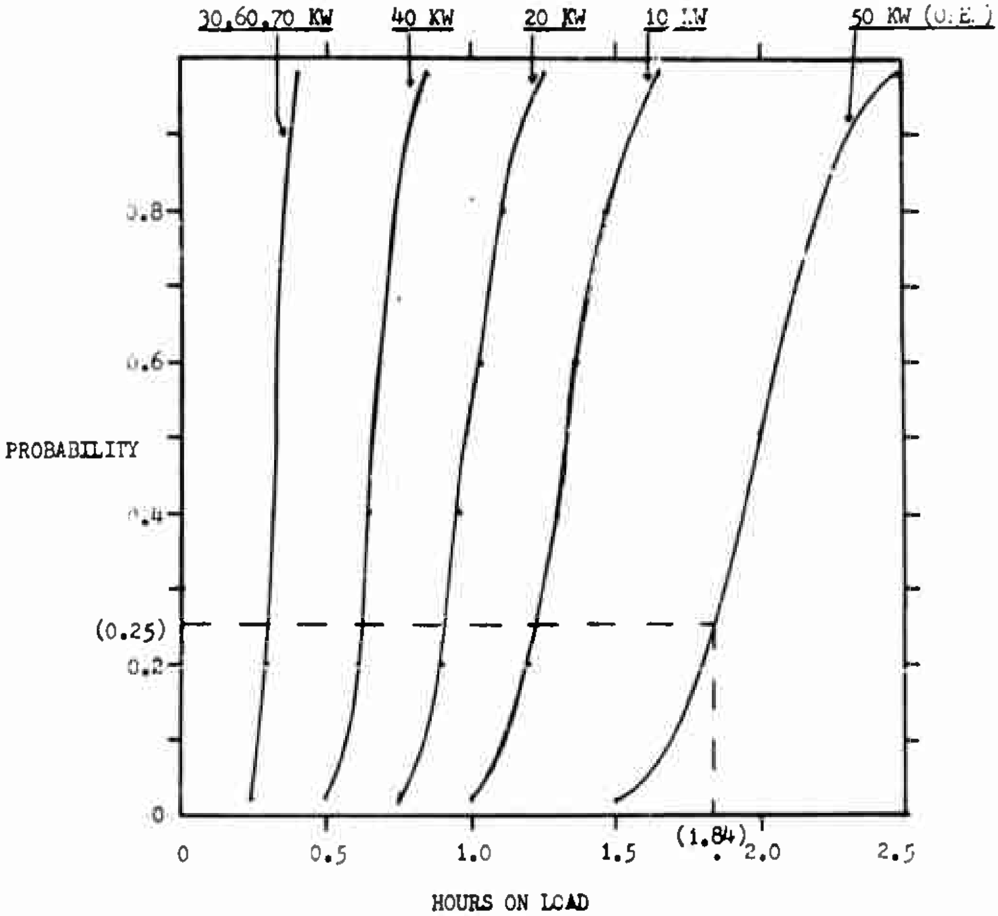


Figure 7. Probability graph of time distribution of loads.

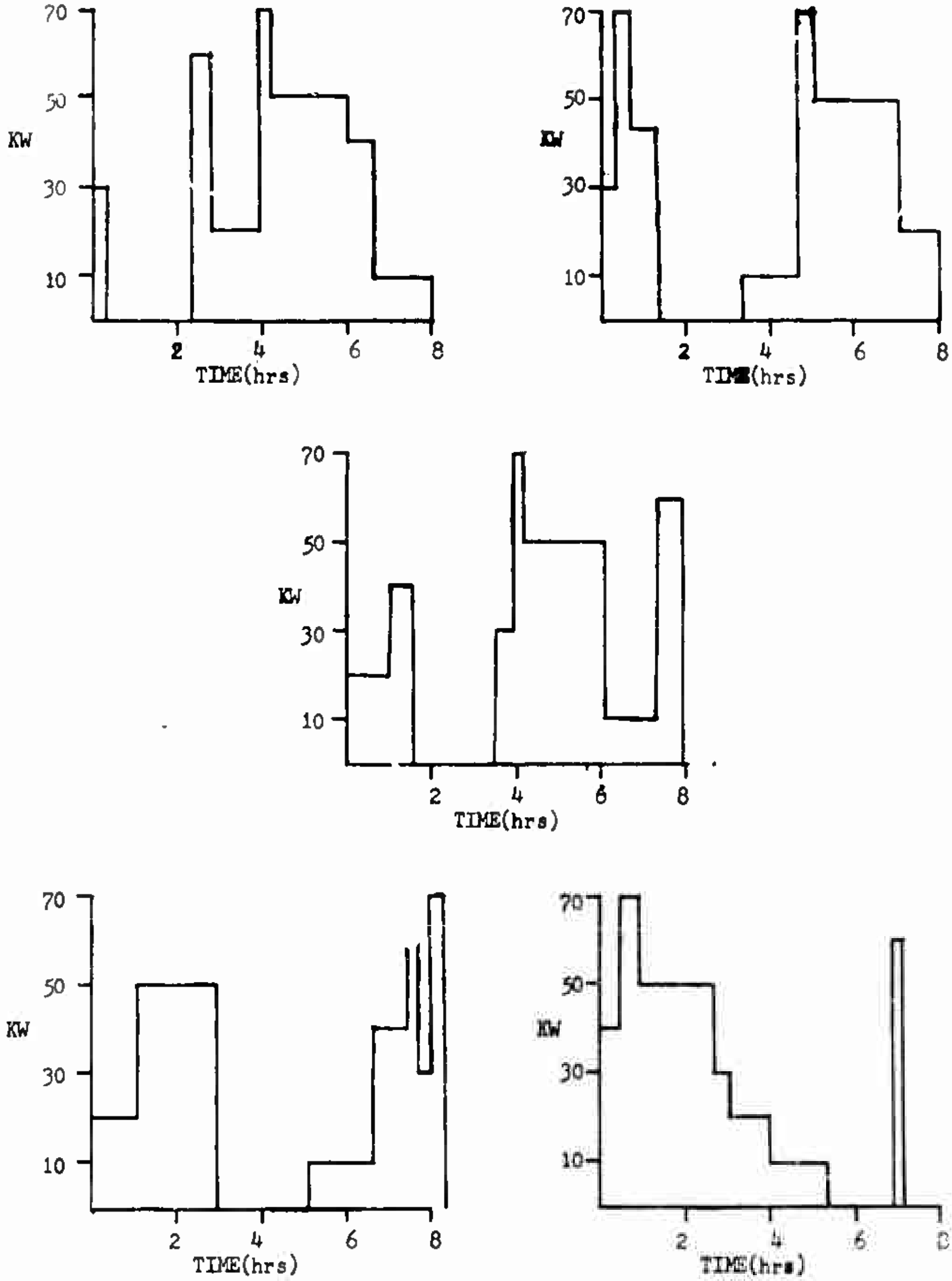


Figure 8. Five load profiles selected at random from 35 sets of random variables

Each of these test load profilee removss approximately the seme emount of power (everage of the five being 205.1 watt-hrs) but in e different sequence of dischargee.

It ie neceesary to collect the teat results into a form which indicatee battery reeponee to the avsrage load profile (Fig. 6) but without a particular discharge eequsnce. Let us assume it ie neceesary to evaluate battery rssponse to a 50 KW load. Thie reeponee will depend on the loads prior to the 50 KW load and on the number of cyclea on the battsry. Since eny number and velue of loade could precede the 50 KW load, all possible prior loede muet be coneidered. Thie is ths sxtremely large number of ell combinations and permutatione of the other seven load values. The battery reeponee to e 50 KW load will then bs the average of the rseponse to all poesible prior loads.

If the five teet load profilae of Fig. 8 are ueed, an approximation to all poeeible loads prior to 50 KW ie made. If the voltage-time curvee of the five load profiles are obtained, the best estimate of the voltage during a 50 KW load will be the average voltags for the 50 KW loed of ths five curves. This seme averaging technique is uaed on all eight load values to give a voltage-time curve after five cyclee which ie independent of discharge sequence, and can be arranged into a voltags reeponee curve corresponding to the power requirements of Fig. 6.

After numerous cycles sre applied to the battery, the data is collected in five cycls sets, and averagee of the reepcnsee to each load are taken. This represente the battery response between, for example, cycles 51 to 55. The eame procedure is continued to compils a voltage-time-cycles contour similar to Fig. 9.

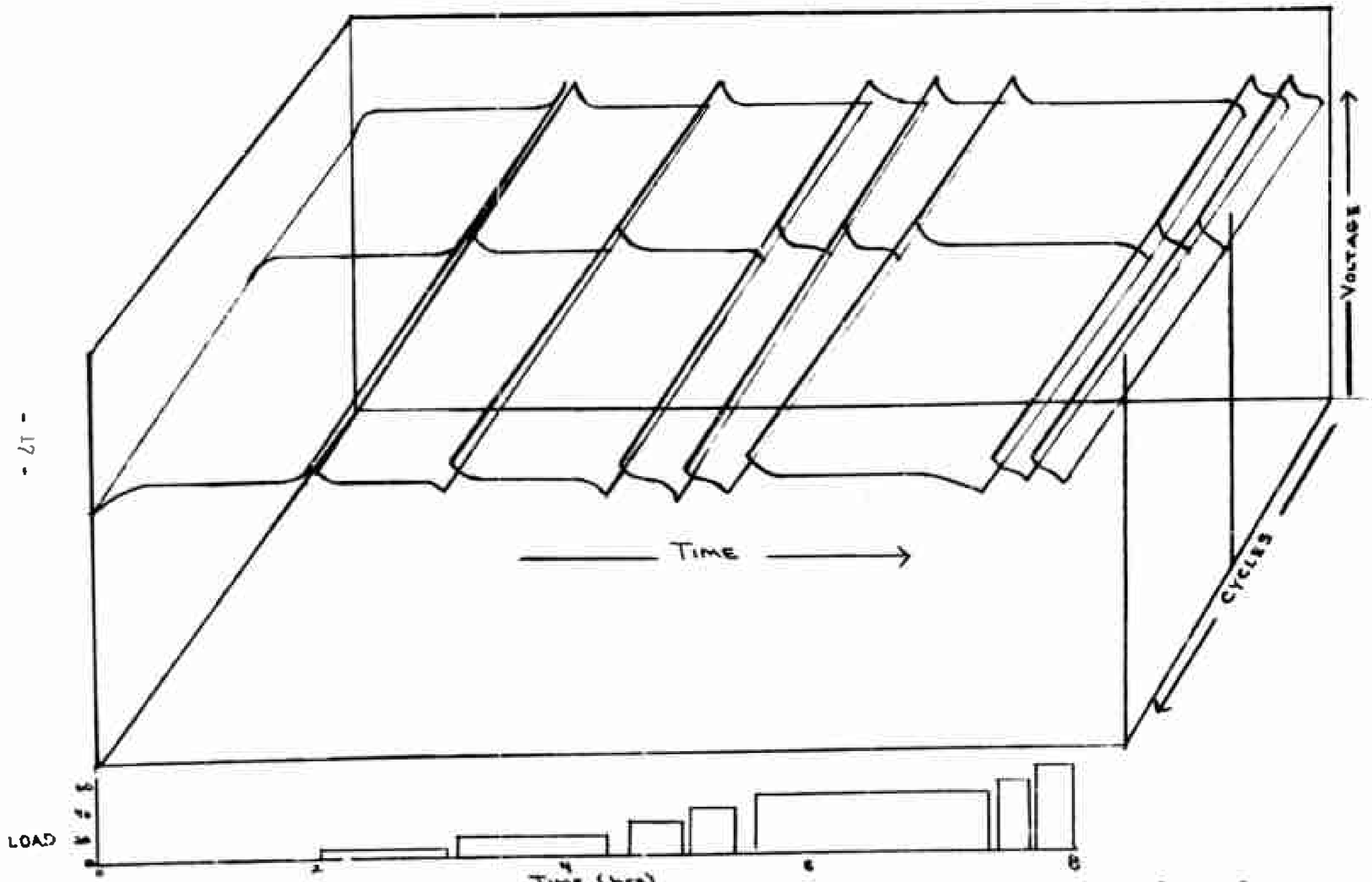


Figure 9. Average voltage - time-cycles surface

The experimental results of battery testing are now in a form in which the results are independent of the discharge sequence and present a more reliable picture of the effect of battery cycling.

Because of the symmetry of voltage-time and current-time curves during constant power discharges, a current-time-cycles contour can be easily constructed from the voltage contour.

## Vehicle Simulation

### A. Discussion

Thus far it has been implied that the power for vehicle propulsion will be supplied solely by batteries which would be recharged at the end of the 8-hour driving period. In a hybrid system, a prime power source is provided in addition to batteries, such that battery power is required only for the higher power demands of the vehicle. At low vehicle power demands, power from the prime source not required for vehicle propulsion is available for battery recharging. A random sequence of the load profile of Fig. 8 applied to a hybrid system would then cause a random mixture of charges and discharges.

It is desired to select a prime power source and a secondary battery such that sufficient power is available to meet the vehicle load profile for all, or nearly all loads. This selection is subject to minimizing the power requirements of the battery for a given prime power source and requiring that the battery be recharged in a reasonable length of time at the end of a driving period.

### B. Results

A 50 KW vehicle is assumed and the load profile of the vehicle is that of Fig. 6. It is necessary to select a prime power source and secondary battery combination which meet the criteria described above. As a first

estimation, a prime power source of 30 KW is assumed. It is then possible to calculate what battery size would be optimal with this size prime power source. With the 30 KW prime source, discharge loads of the battery are then 40 KW, 30 KW, 20 KW, 10 KW; battery charging levels are 30 KW, 20 KW, 10 KW. At the 30 KW vehicle load, the battery is neither charging nor discharging. A Monte Carlo simulation of vehicle operation was made by forming 26 random sequences of the eight level load profile. Calculations were performed on a PDP-8 computer to determine the KWH of charge or discharge applied to or removed from the battery by each step in the sequence, and to determine by a cumulative sum the state of discharge of the battery during the sequence.

Typical of these calculations is as follows:

| Vehicle Load (KW) | Battery* Load (KW) | Time (Hrs) | KWH | Battery* State (KWH) |
|---|---|---|---|---|
| 10 | +20 | 1.33 | 00 | 00 |
| 00 | +30 | 2.00 | 00 | 00 |
| 50 | -20 | 2.00 | -40 | -40.0 |
| 70 | -40 | 0.33 | -13.3 | -53.3 |
| 20 | +10 | 1.00 | +10 | -43.3 |
| 60 | -30 | 0.33 | -10 | -53.3 |
| 30 | 00 | 0.33 | 00 | -53.3 |
| 40 | -10 | 0.67 | -6.7 | -60.0 |

* + = charge, - = discharge

In the particular profile, the first vehicle load is 10 KW, but since 30 KW is available from the prime source, 20 KW of charge is applied to the battery for 1.33 hours. However, as the battery is assumed fully charged at the beginning of the day, no charge is given to the battery, so the

battery state is zero (fully charged). In the third step of the profile 50 Kw is demanded by the vehicle so 20 Kw must be removed from the battery for 2 hours which removes 40 KWH from the battery in this step, leaving the battery state 40 KWH discharged. Similarly, the rest of the table is interpreted.

The data from each of the 86 profiles were examined for the lowest state of discharge in each sequence and the state of discharge at the completion of the 8-hour period. The number of profiles having a given lowest state of battery discharge and the final state of discharge is tabulated (expressed as probability) in Table II.

From the table it is apparent that the battery is most likely to end the 8-hour period in a fully charged state. The next most likely final state of the battery is a 40-50 KWH discharge state. The 40-50 KWH discharge state is also the most likely deepest state of discharge; however, a 60 KWH or greater state of discharge occurs a significant number of times.

The high state of discharge of the battery is less important if the battery does not reach that state until the very end of the profile, as the battery will be recharged regardless. However, from the chart it is apparent that the likelihood of not reaching the lowest state until the end of the profile is quite small.

In 73 of the 86 profiles, a "waste of charge" was involved, i.e., a charge could have been applied to the battery at some step in the profile if the battery were not already fully charged. Sixty-six of these 73 profiles involved a "waste of charge" at other than the last step of the profile. The distribution of the amount of charge wasted during these 66 profiles is:

| Amount of charge "wasted" (KWH) | ≥ 70 | 70-60 | 60-50 | 50-40 | 40-30 | 30-20 | 20-10 | 10-0 |
|---|---|---|---|---|---|---|---|---|
| Fraction of profiles wasting charge of amount: | .06 | .05 | .23 | .21 | .17 | .11 | .14 | .03 |

If this "wasted" charge could have been utilized, e.g., having an auxiliary battery which could always be charged, the lowest state of discharge of the battery would have been:

| State of discharge (KWH) | ≥ 60 | 60-50 | 50-40 | 40-30 | 30-20 | 20-10 | 10-0 | Charged |
|---|---|---|---|---|---|---|---|---|
| Fraction of profiles whose lowest state of discharge is: | .14 | .23 | .29 | .09 | .05 | .15 | .05 | 0 |

TABLE II

FRACTION OF PROFILES VERSUS LOWEST STATE OF DISCHARGE AND FINAL STATE OF DISCHARGE

| State of discharge (KWH) | Fraction of profilss with final state of discharge | Fraction of profilss with lowest state of discharge | Fraction of profiles with lowest state being final state |
|---|---|---|---|
| ≥60 | .05 | .17 | .05 |
| 60-50 | .04 | .33 | .09 |
| 50-40 | .19 | .48 | .16 |
| 40-30 | .07 | 0 | 0 |
| 30-20 | .07 | .01 | 0 |
| 20-10 | .16 | .01 | 0 |
| 10-0 | .09 | 0 | 0 |
| Charged | .26 | 0 | 0 |

Apparently some improvement is made in reducing the lowest state of discharge, e.g., the probability of the lowest discharge being greater than 50KW is decreased from .50 to .37; however, a significant probability exists in having a discherge state greater than 60KW.

C. Conclusions

Simulation involving 86 load profiles of the 81 possible load profiles does not ellow other than the roughest conclusions to be drawn. From this simulation which assumed a 30KW prime power source, it appears that a battery capable of approximately 60 KWH is necessary to meet most vehicle load profile requirements during the 8-hour drive. Further, at the end of the 8-hour period, the battery is likely to be in e cherged state or in a discharge state of less than approximately 40 KWH.

While increasing the power of the prime source will aid in reducing the battery power requirements, it will also increase the likelihood of the battery ending the profile in e fully charged state, implying inefficient use of the battery. It appears then that an optimum selection of prime power source and battery power must exist but further constraints such as cost, weight, etc. must be included. Further refinements in the simulation calculations end inclusion of additional constraints is not likely warranted until a real-time load profile becomes available.

In a similar manner to that eerlier described, five load profiles in which a hybrid configuration is assumed can be selected for battery testing. From the test results voltage-time-cycles contours will be obtained but in addition, overcharge characteristics of the battery can be evaluated.

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13. ABSTRACT This report describes work on two tasks related to improved electrochemical energy conversion systems for vehicle propulsion.

The first is related to the lithium-air cell, specifically the reduction of oxygen at a platinum electrode in 1 M $LiClO_4$ solution in propylene carbonate. Voltage scans in the anodic direction at 200 mv/second gave an oxygen reduction peak at -0.42 volts (versus S.C.E.). An anodic peak at -1.3 volts and a shoulder on a cathodic branch at -2.12 volts had not been observed in the less pure solutions. There is evidence of impurities even in these highly purified solutions.

The second task reported on deals with the mathematical treatment of battery data. The testing of a battery to simulate an 8-hour excursion at various load profiles is described. The treatment is extended to the simulation of vehicle propulsion wherein the battery is associated with a 30 KW prime power source.

Unclassified

Security Classification

| 14. KEY WORDS | LINK A ROLE | LINK A WT | LINK B ROLE | LINK B WT | LINK C ROLE | LINK C WT |
|---|---|---|---|---|---|---|
| Electrochemistry | | | | | | |
| Kinetics | | | | | | |
| Oxygen | | | | | | |
| Non-aqueous Solvent | | | | | | |
| Voltammetry | | | | | | |
| Batteries | | | | | | |

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